Designation: D5263 – 15

Standard Test Method for
Determining the Relative Degree of Oxidation in Bituminous Coal by Alkali Extraction[1]

This standard is issued under the fixed designation D5263; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This colorimetric test method describes the determination of the relative degree of oxidation by alkali extraction of coals that are high volatile A bituminous to low volatile bituminous in rank.

1.2 This test cannot be sensitive to thermally oxidized coal. It is intended for coals that may be oxidized as a result of weathering.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D7448 Practice for Establishing the Competence of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke

3. Terminology

3.1 There are no terms in this standard that require new or other than dictionary definitions.

4. Summary of Test Method

4.1 Humic acids, which are present in oxidized coals, are extracted from the coal with sodium hydroxide solution. The degree of oxidation is determined by colorimetrically measuring the transmittance of the alkali extract solution at 520 nm. The intensity of the color produced by the humic acids is a function of the degree of oxidation.

5. Significance and Use

5.1 This test method is a relative measure of the degree of oxidation present in coal. It does not determine the quantitative amount of oxidized coal present. It is only intended to serve as a guide to the supplier, buyer, and user for selecting coals for metallurgical use.

NOTE 1—Lower rank bituminous coals are more easily extracted than higher rank coal.

6. Apparatus

6.1 *Transmission Spectrophotometer*—Single-beam, grating spectrophotometer having a wavelength range of 340 to 900 nm.

6.2 *Glass Test Tubes/Cuvettes,* with light paths ranging from 10 to 17 mm may be used. Test tubes with an inside diameter of 17 ± 0.3 mm are commonly used.

6.3 *Analytical Balance,* sensitive to 0.001 g.

6.4 *Hot Plate,* capable of bringing aqueous solutions to a boil.

6.5 *Thermometer,* capable of measuring up to 100°C with a sensitivity of ±0.5°C.

6.6 *Filter Papers,* Type II, Class F and G.

6.7 *Timer,* capable of measuring 30 min to the nearest second.

6.8 *Graduated Cylinder,* 100-mL capacity.

6.9 *Beakers,* 400-mL capacity.

7. Reagents

7.1 *Sodium Hydroxide Solution* (certified 1*N*).

7.2 *Octylphenoxypolyethoxyethanol Nonionic Surfactant*—Wetting agent.

7.2.1 Wetting agent shall not be diluted (100 % concentration).

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[1] This test method is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.15 on Metallurgical Properties of Coal and Coke.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 Guidelines for General Acceptance/Rejection Levels of Oxidized Coal

Percent Transmittance at 520 nm, 17-mm Light Path	Interpretation of Results
>90	Coal is not oxidized. Suitable for metallurgical usage.
80 to 90	Coal may be oxidized. If it is being used metallurgically, it should be monitored closely for further changes in oxidation.
<80	Coal is oxidized. Coal at this level may cause coke quality and coke plant operating problems.

8. Calibration and Standardization

8.1 Optical light filters with known absorbance/transmission shall be used to check wavelength accuracy and linearity of the spectrophotometer. Solutions with accurate absorbance values can be found in published literature.[3]

8.2 A blank solution consisting of 100 mL of 1*N* sodium hydroxide is prepared by following the procedure outlined in Section 9, except coal is not used.

9. Procedure

9.1 Activate the spectrophotometer and set at 520-nm wavelength; allow sufficient time for the instrument to stabilize (30 min).

9.2 Weigh out 1 g (±0.01 g) of the −250-µm (No. 60 U.S. Standard Sieve Series) coal sample.

9.3 Transfer the coal to a standard 400-mL beaker.

9.4 Add 100 mL of NaOH solution and one drop of wetting agent (100 % concentration) to the coal. Place a glass stirring rod into the beaker.

NOTE 2—Wetting agent drop size can significantly influence results, with larger drop size causing lower transmittances.

9.5 Place beaker(s) onto a preheated hot plate and place a thermometer in the beaker.

9.6 Bring temperature of the contents to 98°C. It shall take 3 to 4 min to reach a boil, depending on the number of beakers on the hot plate. When measuring the temperature of the blank solution or test solution, or both, suspend the thermometer in a manner such that the temperature of the solution and not the temperature of the beaker resting on top of the hot plate is being measured.

9.7 At the time the blank solution or test solution, or both, in the beakers reaches the temperature of 98°C, set the timer for 3 min. Allow the solution to boil for 3 min, stirring the contents for at least 5 s for each 1-min interval.

9.8 After the 3-min boil, carefully remove hot beakers and allow to cool under ambient conditions for 30 min. This is also determined by using a timer.

9.9 Filter (by gravity) the entire blank solution or test solution, or both, on a stack of one Type II Class F filter paper on top of one Type II Glass G filter paper. Filter the slurry into a 100-mL graduated cylinder, making sure funnel and graduated cylinder are dry before each test. After filtration, add deionized (distilled) water (Type II, Specification D1193) to the cylinder to bring the solution to 80 mL and stir with a glass rod.

NOTE 3—The beaker should not be rinsed onto the filter paper. It is not significant if some coal remains in the beaker.

9.10 Adjust the 0 and 100 % transmittance levels of the spectrophotometer.

9.10.1 Adjust the spectrophotometer to read zero by closing the lid of the sample/tube holder assembly of the instrument.

9.10.2 Fill cuvette/test tube with the blank solution and insert into the spectrophotometer.

9.10.3 Adjust the spectrophotometer to read 100 % transmittance by inserting the blank solution (see 8.2).

9.10.4 Periodically during the analysis, check the span (0 and 100 % transmittance) to be sure that the instrument has not drifted.

9.11 Place the cuvette/test tube containing the test solution into the spectrophotometer; measure and record the percent transmittance of each test solution. Use only cuvette/test tubes for the test solution that yield the same transmittance values as the cuvettes/test tubes used with the blank solutions.

10. Calculation

10.1 Since the light path is a variable in this test, results must be adjusted to a common basis to make comparisons. A 17-mm light path will be used. Thus, if the test was performed using any other light path than 17 mm, the result must be adjusted to the 17-mm light path using Beer's law.

10.2 Adjust results as follows:

$$A_x = -\mathrm{Log}\left(\frac{T_x}{100}\right) \tag{1}$$

$$A_{17} = A_x(17\ \mathrm{mm}/b_x) \tag{2}$$

$$T_{17} = \left(10^{-A_{17}}\right) \times 100 \tag{3}$$

where:

T_x = transmittance obtained by analysis, %;
A_x = absorbance corresponding to T_x;
b_x = test light path, mm;
A_{17} = absorbance at 17-mm light path; and
T_{17} = transmittance (calculated) at 17-mm light path.

11. Report

11.1 Report the value T_{17} (obtained in Section 9).

11.2 All accountability and quality control aspects of Guide D7448 apply to this test method.

12. Precision and Bias

12.1 *Precision*—The relative precision of this test method for the determination of the degree of oxidation of bituminous coal covers the range (percent transmittance) from 55 to 98 %.

12.1.1 *Repeatability*—The difference in absolute values between two consecutive tests conducted on the same sample in the same laboratory by the same operator using the same apparatus should not exceed the repeatability interval *I*(*r*) more

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[3] Mellon, M. G., *Analytical Absorption Spectroscopy*, John Wiley & Sons, Inc., New York, 1950, pp. 258–265.

than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the repeatability interval, there is reason to question one or more of the test results. The repeatability interval for this test method is 3.0 % transmittance at the standard 17-mm light path.

NOTE 4—Duplicate determinations are not required by this test method. The repeatability of the test has been determined for informational purposes and for those that require this information for internal quality control. Most internal quality control programs require a known acceptance value for duplicate determinations.

12.1.2 *Reproducibility*—The difference in absolute value of replicate determinations, conducted in different laboratories on representative samples prepared from the same bulk sample after the last stage of reduction, should not exceed the reproducibility interval *I*(*R*) more than 5 % of such paired values (95 % confidence level). When such a difference is found to exceed the reproducibility interval, there is reason to question one or both of the test results. The reproducibility interval for this test method is 7.6 % transmittance at the standard 17-mm light path.

12.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method for measuring oxidation of coal, bias has not been determined.

13. Keywords

13.1 absorbance; alkali extraction; colorimetric; humic acids; oxidation (weathering); transmittance

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